

# SCIENCE FOR GLASS PRODUCTION

UDC 666.171:666.172.6:536.5:539.4:31:666.1

## STATISTICAL EVALUATION OF THE EFFECT OF THE MOLD TEMPERATURE ON THE STRENGTH OF GLASS CONTAINERS

N. Yu. Mikhailenko,<sup>1</sup> P. D. Sarkisov,<sup>1</sup> and L. I. Mangutova<sup>1</sup>

Translated from *Steklo i Keramika*, No. 2, pp. 3–7, February, 2007.

Using methods of mathematical statistics (cluster, dispersion, and correlation analyses), the impact strength of three types of glass containers (bottles and jars) was analyzed. Three temperature regions in which molding results in very different values of the strength of the articles are revealed. The boundaries of the molding temperature regions are critical regardless of the type of article molded and are probably only determined by the chemical composition of the glass melt. The preferred mold temperature range is 480–550°C. The results of the study are interpreted based on an analysis of the features of molding glass at different temperatures.

Strength is the most important performance characteristic of the glass container. In conditions of industrial production, the molding conditions have a large effect on the strength of glassware. The strength of glass containers processed at Kamyshin Glass Container Plant was investigated as a function of the mold temperature in the present study.

The studies were conducted on bottles and jars of different capacity processed by double blowing and press and blow methods in sectional glass molding machines (Table 1). All articles were processed from sodium–calcium–silicate glass melt melted according to the same formulation. The glass gob temperature varied in the 1110–1130°C range as a function of the article type. Kleenmold lubricant was used for lubricating the molds. The mold temperature was deter-

mined with a Handy-2MEM contact pyrometer from Rondot with accuracy of  $\pm 1^\circ\text{C}$ . The range of variation of the mold temperature was 430–650°C. The articles were annealed and controlled in accordance with the technical regulations and process conditions for the finished product used in the plant.

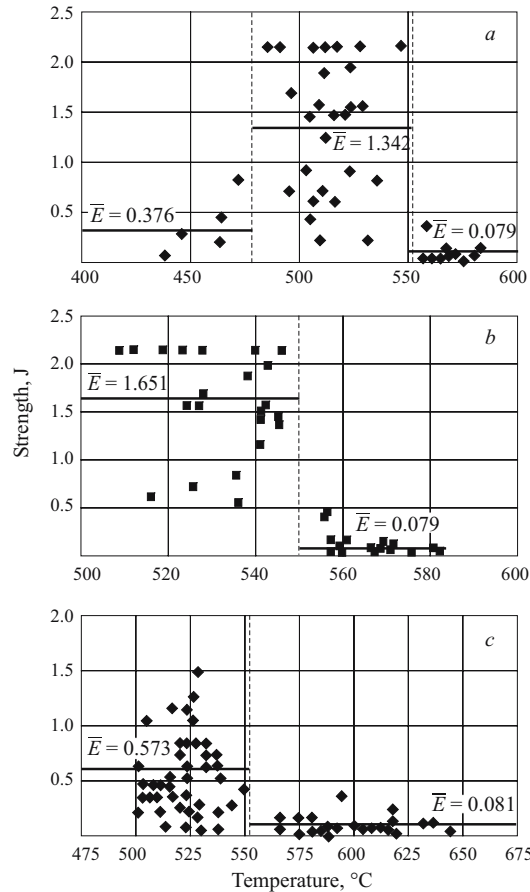
The impact strength was determined to characterize the mechanical properties of the glass containers (Fig. 1). We know that an impact load is especially dangerous for glass, since it creates higher mechanical stresses in glass than the same load applied slowly (static load). The impact resistance of glass reflects its most typical property — the brittleness. The tests were performed on a standard impact hammer.

Methods of mathematical statistics — cluster, dispersion, and correlation analysis — were used to evaluate and analyze the results of the tests. The statistical calculations were performed with Microsoft Excel and SPSS software.

<sup>1</sup> D. I. Mendelev Russian Chemical Engineering University, Moscow, Russia; Kamyshin Glass Container Plant, Volgograd Region, Russia.

TABLE 1

Characteristics of articles	K128 bottles	K114 bottles	1-82 jars
Brand	K128-KV-2-83-250	K114-V43A-750	1-82-3000
Capacity, liters	0.25	0.75	3.00
Molding method	Double blowing		Press and blow
Glass-molding machine	6-section with two-seat molds		8-section with one-seat molds
Gob temperature, °C	1125	1130	1110
Temperature of clean molds, °C	430–590	500–590	500–650



**Fig. 1.** Impact strength of K128 (a) and K114 bottles (b) and 1-82 jars (c) processed at different mold temperatures.

A cluster analysis of the strength values within the limits of each type of article was conducted in the first stage. The essence of cluster analysis consists of procedures for separating the variable value of the individual clusters from a set of values based on some trait [1], in the given case, based on the values of the strength and its dispersion.

The results of cluster analysis showed that in each group of articles, the entire set of strength values can be divided into several clusters corresponding to the different temperature regions of molding the articles. In the case of K128 bottles, three clusters corresponding to three molding temperature regions were distinguished — low-temperature (“cold” molds), regions of medium and high temperatures (“superheated” molds). The set of strength values for K114 bottles and 1-82 jars was divided into two clusters corresponding to medium molding temperatures and the high-temperature region. The low-temperature molding region is missing, since these articles were not processed in “cold” molds.

A total of seven clusters or samples (“sample” means the set of values of the impact strength of articles from each of the three indicated types molded in different temperature regions). It is especially important to note that the boundaries of the detected temperature regions almost coincide in all groups of articles and are approximately 480°C (low-temper-

ature region — medium temperature region) and approximately 550°C (high-temperature region — medium temperature region). As a consequence, the boundaries of the molding temperature regions are critical regardless of the type of article molded are probably determined by the chemical composition of the glass melt alone.

In molding in “cold” molds, the impact strength  $E$  is characterized by the average value of 0.57 J (K128 bottles). Molding in the medium-temperature region causes high scattering of the strength values, but the maximum values (2.15, 2.15, and 1.48 J for K128, K114, and 1-82 articles, respectively) were obtained together with the low values in this region for all types of articles. Molding in “superheated” molds sharply reduced the strength of all types of articles — the minimum strength values were obtained in this temperature region.

In the second stage, the experimental data obtained were statistically tested for the homogeneity of the sampling data (testing the samples for the presence of outliers, i.e., “gross” errors) and the correspondence of the sampling data in each sample to the law of normal distribution (dispersion analysis is used if the law of normal distribution of the random quantity investigated is satisfied), and they were evaluated by methods of dispersion analysis. To verify the “gross” errors, we used the normalized distribution of the maximum relative deviations at the 95% confidence level [2]. One strength value was excluded from each of the three samples as “gross” errors. The statistical description of the samples after exclusion of the “gross” errors is presented in Table 2.

The correspondence of the distribution of the strength values in the samples to normal distribution was verified by comparing the asymmetry  $A$  and excess  $E$  parameters of the samples (see Table 2) with their representativeness errors  $m_A$  and  $m_E$ . The representativeness errors were calculated with the equations:

$$m_A = \sqrt{\frac{6}{n}};$$

$$m_E = 2 \sqrt{\frac{6}{n}},$$

where  $n$  is the number of sampling data in the sample; the distribution is considered normal if the inequalities  $A < 3m_A$  and  $E < 3m_E$  are satisfied.

The calculations showed that in each sample, the distributions of the strength values correspond to normal distribution.

The dispersion analysis was conducted to compare the samples with each other with respect to their statistical parameters — dispersion of strength  $s^2$  and average strength  $\bar{E}$  (see Table 2). Such a comparison allows validly accepting or rejecting the hypothesis that the compared samples belong to the same general set of strength values (“null hypothesis”) [2]. Since the samples include the experimental values of the strength of different articles molded in different temperature

TABLE 2

Statistical strength parameters	K128 bottles			K114 bottles		1-82 jars	
	low-temperature molding region (438 – 472°C)	average temperatures (485 – 546°C)	high-temperature molding region (557 – 582°C)	average temperatures (509 – 546°C)	high-temperature molding region (556 – 582°C)	average temperatures (501 – 549°C)	high-temperature molding region (566 – 645 °C)
Number of tests	5	28	10	23	12	48	28
Strength, J:							
minimum	0.08	0.23	0.02	0.54	0.02	0.04	0.02
maximum	0.83	2.15	0.17	2.15	0.17	1.48	0.23
range of variation	0.75	1.92	0.15	1.61	0.15	1.44	0.21
sum of values	1.88	37.58	0.79	37.97	0.95	27.49	2.28
average value	0.376	1.342	0.079	1.651	0.079	0.573	0.081
Sampling dispersion	0.082	0.462	0.002	0.299	0.002	0.110	0.003
Standard deviation	0.286	0.680	0.047	0.547	0.042	0.332	0.054
Standard error	0.128	0.128	0.015	0.114	0.012	0.048	0.010
Median	0.29	1.48	0.08	1.69	0.08	0.54	0.08
Mode	–	2.15	0.04	2.15	0.08	0.37	0.04
Excess	1.430	– 1.331	– 0.086	– 0.503	0.537	0.010	0.757
Asymmetry	1.141	– 0.268	0.689	– 0.837	0.718	0.660	1.087

regions, the above equation should confirm or disprove the hypothesis concerning the effect of these variable factors on the impact strength of the articles. If  $\bar{E}$  and  $s^2$  of different samples differ statistically insignificantly (at the selected significance level), then we can hypothesize that the samples belong to the same general set and scattering of the strength values in these samples will be determined by random errors or unconsidered factors, i.e., the strength will not be a function of the type of article and the molding temperature. If parameters  $\bar{E}$  and  $s^2$  of different samples differ statistically significantly, then the effect of the type of article and/or the molding temperature parameters on the strength characteristics of the articles must be objectively identified.

The strength dispersions were compared with Fisher's  $F$  test by unifactorial dispersion analysis, and different combinations of samples were tested. The calculated values of the  $F$  of the samples were compared with the critical values of

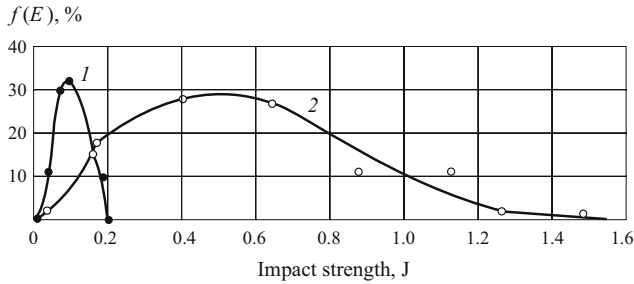
$F(f_1; f_2)$  of known numbers of degrees of freedom  $f$  within samples and between samples and 0.05 significance level (95% confidence level). The average strength values  $\bar{E}$  were compared with Student's  $t$  test at the same significance level in the condition of statistical equality of their dispersions.

The initial data and results of the dispersion analysis and  $t$ -test on the samples are reported in Table 3. In all cases, the dispersions of the samples related to one type of article but molded in different temperature regions differ statistically significantly, i.e., these samples cannot be combined in a common set. The results of cluster analysis, which allows dividing the indicated molding temperature ranges into independent regions, were thus confirmed by the data from dispersion analysis.

The most interesting results were obtained in analyzing samples belonging to different types of articles molded in the same temperature regions — high-temperature and me-

TABLE 3

Molding temperature region	K114 bottles	k128 bottles	1-82 jars	Results of dispersion analysis and $t$ -test
High-temperature	$s^2 = 0.002$ $f = 11$ $\bar{E} = 0.079$	$s^2 = 0.002$ $f = 9$ $\bar{E} = 0.079$	$s^2 = 0.003$ $f = 27$ $\bar{E} = 0.081$	$F = 0.01 < F_{0.95}(2; 47) = 3.1$ 9 (dispersions equal) $ t  = 0.13 < t_{0.95}(18) = 2.1$ 0 (averages equal)
Medium temperatures	$s^2 = 0.299$ $f = 22$	$s^2 = 0.462$ $f = 27$	$s^2 = 0.110$ $f = 47$	$F = 42.8 > F_{0.95}(2; 96) = 3.1$ (dispersions not equal)
Low-temperature	–	$s^2 = 0.082$ $f = 4$	–	–
Result of dispersion analysis	$F = 97.39 > F_{0.95}(1; 33) = 4$ . $F = 21.10 > F_{0.95}(2; 40) = 3$ . $F = 60.12 > F_{0.95}(1; 74) = 3$ . 14 (dispersions not equal)    23 (dispersions not equal)    97 (dispersions not equal)			–



**Fig. 2.** Impact strength distribution functions for articles molded in different temperature regions: 1 and 2) high and medium mold temperatures, respectively.

medium-temperature regions. The results of the analysis indicate that the dispersions and average values for samples of all kinds of articles can be considered statistically equal and belonging to one general set in molding in the high-temperature region. In this case, the article type does not affect the impact strength; the strength is only determined by the fact that the articles were molded in “superheated” molds.

In molding in the medium-temperature region, another picture is observed — articles of the three different types cannot be combined in one set since their dispersions differ statistically significantly.

Articles (medium temperature region)	Dispersion analysis and $t$ -test results
K128 and K114 bottles. . . . .	$F = 3.09 < F_{0.95}(1; 49) = 4.04$ (dispersions equal) $ t  = 1.80 > t_{0.95}(49) = 1.68$ (average values not equal)
K114 bottles, 1-82 jars . . . . .	$F = 106.09 > F_{0.95}(1; 69) = 3.98$ (dispersions not equal)
K128 bottles, 1-82 jars . . . . .	$F = 43.89 > F_{0.95}(1; 74) = 3.97$ (dispersions not equal)

Paired analysis of the samples showed that both kinds of bottles can be combined with respect to the equality of dispersions trait, although the average strength values for them differ statistically significantly. At the same time, none of the bottle types can be combined with the jars, which are an independent sample. The experimental strength distribution functions calculated for the high-temperature region for a general set of three samples and for the medium-temperature region for the set for 1-82 jars are shown in Fig. 2.

Although molding of any articles in “superheated” molds results in the same low strength values regardless of the arti-

cle type, molding of bottles and jars in the medium-temperature region differs significantly, which could be due to different molding methods (see Table 1). If the articles are molded by the same method (bottles), then the average values will be determined by the configuration and capacity of the concrete article type for the same scattering of the strength values.

In the concluding stage of the study, we assessed the correlation between the impact strength of the articles and their molding temperature (mold temperature) in each temperature region. The correlation coefficients  $r$  were calculated with the standard method of correlation analysis and their significance was evaluated by comparison with the critical values of  $r_{(f)}$  ( $r$ -distribution) for the 95% confidence level for  $f$  sample degrees of freedom [2]. In all cases, the correlation coefficients have low values statistically indistinguishable from zero (Table 4). There is thus no correlation between the mold temperature and article strength within the same temperature region.

We know that the strength characteristics of glassware are primarily determined by the defectiveness of their surface layers — by the presence of dangerous microcracks, crazes, and other defects formed during molding and subsequent use of the article. For this reason, interpretation of the results obtained in the present study should be based on an analysis of the features of molding the articles in the different temperature regions (at different mold temperatures).

In molding, when the glass melt gob comes into contact with the mold (shaping stage), the hot surface layers of the gob cool abruptly so that they harden. The hardening rate has a determining effect on both the molding process and the surface state of the glassware [3].

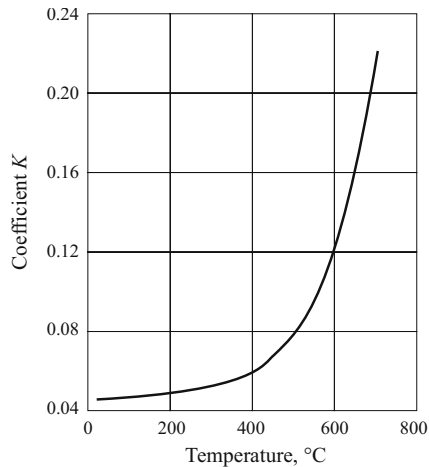
In molding in “cold” molds, such a high temperature gradient arises between mold and gob and inside the gob that the thin surface layer of the molded article is destroyed, with formation of microcracks and crazes due to the insufficient thermal stability of the glass. This is determining in molding of glass in “cold” molds, so that the strength of the articles in this case cannot be high, but the dispersion of the strength values is low.

When the mold temperature increases (medium-temperature region), the temperature gradient between gob and mold and over the thickness of the article decreases, which causes the surface defectiveness to decrease. This temperature region is best for molding, since the maximum strength values can be attained in it. However, very high dispersion of the strength values is observed here, which indicates the effect of other factors not taken into account. In addition, in the indicated temperature region, the effect of the molding method and the configuration and capacity of the articles (scale factor) is manifested. The range of this region is 480 – 550°C. The upper limit corresponds to the glass transition temperature  $t_g$  of the glass.

When the molding temperature is above  $t_g$ , the strength of all types of articles decreases sharply with low dispersion of its values. This is probably due to an important change in

**TABLE 4**

Molding temperature region	Correlation coefficient $r$		
	K114 bottles	k128 bottles	1-82 jars
High-temperature	– 0.440	0.280	– 0.160
Medium temperatures	– 0.190	– 0.002	0.050
Low-temperature	–	– 0.003	–



**Fig. 3.** Coefficient  $K$  as a function of the temperature of the external (contact) cooling medium.

the thermophysical properties of the glass in going into the region of the viscoplastic state and correspondingly, a change in the cooling and hardening conditions. It was experimentally shown in [4] that increasing the temperature of the external (contact) cooling medium above the glass transition temperature causes a sharp increase in coefficient  $K$ , characterizing the glass melt cooling rate (Fig. 3):

$$K = \frac{SC}{mc_p},$$

where  $S$  and  $m$  are the surface area and mass of the article, respectively;  $C$  is the radiation coefficient, or radiation constant;  $c_p$  is the specific heat capacity of the glass.

An increase in the cooling intensity in turn causes an increase in the defectiveness of the surface layers of the article. In addition, with a further increase in the mold temperature, the glass melt can begin to stick to the walls of the mold [5]. The minimum dispersion of the strength of articles molded in “superheated” molds indicates that in the given case, the temperature factor is prevalent, and low strength values are primarily determined by the properties of the glass melt itself.

The analysis of the strength characteristics of glass containers using methods of mathematical statistics thus allowed distinguishing three temperature regions in which molding results in significantly different strengths for the glassware, and determining the preferred temperature region consisting of 480 to 550°C regardless of the type of molded item and the molding method.

## REFERENCES

1. B. S. Duran and P. L. Odell, *Cluster Analysis: A Survey in: Lecture Notes in Economics and Mathematical Systems*, No. 100, Springer, Berlin (1974).
2. E. I. Pustyl'nik, *Statistical Methods of Analysis and Processing of Sampling Data* [in Russian], Nauka, Moscow (1968).
3. V. P. Khramkov, Yu. A. Guloyan, and V. I. Laptev, *Molding of Glassware* [in Russian], Legkaya Industriya, Moscow (1980).
4. I. I. Kitaigorodskii and S. I. Sil'vestrovich (eds.), *Glass Production Handbook* [in Russian], Stroiizdat, Moscow (1963).
5. R. Bruckner and H. Jebsen-Marwedel (eds.), *Glastechnische Fabrikationsfehler [Manufacturing Defects in Industrial Glass]*, Springer-Verlag, Berlin (1980).